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National Food Safety Standard-Trisodium Phosphate

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Report Highlights:

On May 5, 2010, China notified the WTO of National Food Safety Standard: Food Additives – Trisodium Phosphate as SPS/N/CHN/243. This measure applies to the production, circulation, supervision and management of the food additive Trisodium Phosphate. It specifies the scope, requirements and testing methods. The date for submission of final comments to China was May 20, 2010. The proposed date of entry was May 30, 2010. Contact information on where to send comments is inside the report. This report is an INFORMAL translation of this document.

Executive Summary:

On May 5, 2010, China notified the WTO of National Food Safety Standard: Food Additives – Trisodium Phosphate as SPS/N/CHN/243. This measure applies to the technical requirements and characterization of the food additive Trisodium Phosphate. It specifies the scope, requirements and testing methods. The date for submission of final comments to China was May 20, 2010. The proposed date of entry was May 30, 2010.

Comments can be sent to the China WTO SPS Enquiry Point at: SPS@aqsiq.gov.cn.

This report contains an UNOFFICIAL translation of National Standard on Determination of Trisodium Phosphate in Foods.

General Information:

BEGIN TRANSLATION

**National Food Safety Standard
Food Additive-Trisodium Phosphate**
(Draft for soliciting opinions)

Promulgated on xx-2010
Executed from xx-201X

Foreword

For revision of this standard (the Standard), "Trisodium Phosphate" of the 6th version of American standard Food Chemicals Codex (FCC 6) is adopted.

The technological disparities between the Standard and "Trisodium Phosphate" of FCC 6 include:

- Content and indicator and testing method for heavy metal is added;
- Quimociac method gravimetric method is apposed in the testing method for content of trisodium phosphate;
- For the testing method for arsenic content, diethyl dithio carbamic acid – limited Ag colorimetric method is adopted in FCC, whereas determination of arsenic in food additive, i.e. arsenic stain method is adopted in the Standard.

Appendix A to the Standard is a normative appendix.

**National Food Safety Standard
Food Additive Trisodium Phosphate**

1. Scope

The Standard shall be applicable for trisodium phosphate, a kind of food additive produced by thermal processing of phosphoric acid and industrial soda or ion film caustic soda as raw material.

2. Cited normative documents

The documents cited in the Standard are imperative for application of the Standard. For the cited

documents with a date, only the version of the date shall be applicable for the Standard; for those without date, the latest version (including all revision lists) shall be applicable for the Standard.

3. Molecular formula and relative molecular mass

3.1 Trisodium phosphate anhydrous

Molecular formula: Na_3PO_4

Relative molecular mass: 163.94 (as per international relative atomic mass in 2007)

3.2 Trisodium phosphate monohydrate

Molecular formula: $\text{Na}_3\text{PO}_4 \cdot \text{H}_2\text{O}$

Relative molecular mass: 181.96 (as per international relative atomic mass in 2007)

3.3 Trisodium phosphate dodecahydrote

Molecular formula: $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$

Relative molecular mass: 380.18 (as per international relative atomic mass in 2007)

4. Technical requirements

4.1 Requirements for evaluation: shall comply with the regulations of Table 1.

Table 1 Requirements for sense evaluation

Item	Requirements	Testing method
Color	White	Take an amount of sample and put it in a beaker of 50ml, and observe the color and textural state in natural light.
Textural state	Crystal or powder	

4.2 Physical and chemical indicator: shall comply with the regulations of Table 2.

Table 2 Physical and chemical indicators

Item	Indicator	Testing method
Trisodium phosphate anhydrous (Na_3PO_4) (Calculated as per ignited dry basis, Na_3PO_4), w/% Trisodium phosphate monohydrate ($\text{Na}_3\text{PO}_4 \cdot \text{H}_2\text{O}$) (Calculated as per ignited dry basis, Na_3PO_4), w/% Trisodium phosphate dodecahydrote ($\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$), w/%	\geq \geq \geq 97.0	A.4 of Appendix A
pH value (10g/l solution)	11.5~12.5	A.5 of Appendix A
Arsenic (As), w/%	\leq 0.0003	A.6 of Appendix A
Fluorine (F), w/%	\leq 0.005	A.7 of Appendix A
Heavy metal (as per Pb), w/%	\leq 0.001	A.8 of

			Appendix A
Lead (Pb), w/%	≤	0.0004	A.9 of Appendix A
Water insoluble substance, w/%	≤	0.2	A.10 of Appendix A
Ignition loss (Na ₃ PO ₄), w/%	≤	2	A.11 of Appendix A
(Na ₃ PO ₄ •H ₂ O), w/%		8~11	
(Na ₃ PO ₄ •12H ₂ O), w/%		45~57	

Appendix A Testing method (normative appendix)

A.1 Cautions

The reagent applied in this testing method is toxic and corrosive, so care shall be taken in operation! If necessary, the test may be conducted in fume cupboard. If any reagent spatters on skin, rinse it with water immediately, or go to hospital for treatment if serious.

A.2 General conditions

Except otherwise specified, reagent and water applied in the Standard shall be pure reagent and water in Class III as specified in GB/T 6682-2008, and the required standard solution and that of impurity, agent and product shall be prepared in accordance with HG/T 3696.1, HG/T 3696.2, and HG/T 3696.3.

A.3 Identification

A.3.1 Reagent and material

A.3.1.1 Hydrochloric acid (HCL);

A.3.1.2 Acetic acid solution: 1+1;

A.3.1.3 Ammonia solution: 2+3;

A.3.1.4 Silver nitrate solution: 17g/l;

A.3.1.5 Platinum wire loop

A.3.2 Identification test

A.3.2.1 Identification of sodium ion

Take and weigh 1g of sample and add in 20ml of water for dissolving, and use platinum wire loop and dip HCl for wetting; and then, burn it on flame to colorless; dip again the test solution and burn on the flame, now the flame should be in yellow.

A.3.2.2 Identification of phosphate radical ion

Take and weigh 0.1g of sample and dissolve it in 10ml of water, and add 1ml of silver nitrate solution to generate yellow deposit. The deposit can dissolve in ammonia solution but not in acetic acid solution.

A.4 Determination of trisodium phosphate content

A.4.1 Quimociac gravimetric method (arbitration method)

A.4.1.1 Method summary

In acid medium, use Quimociac precipitant in the test solution to enable the phosphate radical to form into Quinoline phosphomolybdate deposit fully; after filter, dry, and weigh the deposits, calculate the content of trisodium phosphate in the sample.

A.4.1.2 Reagent

A.4.1.2.1 Nitric acid;

A.4.1.2.2 Nitric acid solution: 1 + 1;

A.4.1.2.3 Quimociac solution

Quimociac solution prepared in accordance with the requirements of HG/T 3696.3 is stored in polyethylene bottle.

A.4.1.3 Instrument and equipment

A.4.1.3.1 Sintered glass crucible: filter board aperture is $5\mu\text{m}\sim 15\mu\text{m}$;

A.4.1.3.2 Electrothermal constant temperature dry box: the temperature can be controlled at $180^{\circ}\text{C}\pm 5^{\circ}\text{C}$ or $250^{\circ}\text{C}\pm 10^{\circ}\text{C}$.

A.4.1.4 Analysis steps

A.4.1.4.1 Preparation of test solution

Take and weigh about 2g of sample of Na_3PO_4 or $\text{Na}_3\text{PO}_4\cdot\text{H}_2\text{O}$ ignited as specified in A.11 ($\text{Na}_3\text{PO}_4\cdot 12\text{H}_2\text{O}$ shall be the original sample), and keep the accuracy to 0.0002g; put in a beaker of 100ml, dissolve and transfer all into a measuring flask of 500ml, and dilute with water to the scale and shake up.

A.4.1.4.2 Determination

Use a pipette and take out 15ml of test solution into a beaker of 250ml, and add 10ml of nitric acid solution and water to a bulk volume of about 100ml; add 50ml of Quimociac solution and cover the watch glass, heat up on hot plate or in water bath to a room temperature of $75^{\circ}\text{C}\pm 5^{\circ}\text{C}$, and keep for 30s (no open flame shall be used for heating up, and no mixing shall be permitted when adding reagent or heating up, so as to avoid generation of irregular nodule). Cool to room temperature, and mix round for 3~4 times during cooling. Filter with sintered glass crucible dried to constant mass at $180^{\circ}\text{C}\pm 5^{\circ}\text{C}$ or $250^{\circ}\text{C}\pm 10^{\circ}\text{C}$. Firstly filter the clear liquid in the top layer, and wash and deposit for 2 times by decantation method, for which the water used is about 20ml each time; transfer the deposit into sintered glass crucible, wash the inner wall of the crucible with water continuously and deposit for several times, for which the water used is about 150ml totally. And then, put the crucible in the electrothermal constant temperature dry box, dry for 45min at $180^{\circ}\text{C}\pm 5^{\circ}\text{C}$ or 15min at $250^{\circ}\text{C}\pm 10^{\circ}\text{C}$; take out and put into drier and cool to room temperature, weigh and keep an accuracy of 0.0002 g.

Simultaneously make blank test, during which no sample shall be added in the test solution, but others shall be the same, such as type and amount of reagent.

A.4.1.5 Calculation

The content of Na_3PO_4 or $\text{Na}_3\text{PO}_4\cdot\text{H}_2\text{O}$ shall be calculated as per the mass fraction of Na_3PO_4 (w_1) and be expressed by % as shown in Formula A1:

$$w_1 = \frac{(m_1 - m_2) \times 0.07408}{m \times \frac{15}{500}} \times 100 \dots\dots\dots (\text{A.1})$$

The content of $\text{Na}_3\text{PO}_4\cdot 12\text{H}_2\text{O}$ shall be calculated as per the mass fraction (w_2) and be expressed by % as shown in Formula A2:

$$w_2 = \frac{(m_1 - m_2) \times 0.1718}{m \times \frac{15}{500}} \times 100 \dots\dots\dots (\text{A.2})$$

In which:

m_1 -- Mass of Quinoline phosphomolybdate deposit generated from the test solution (unit: g);

m_2 -- Mass of Quinoline phosphomolybdate deposit generated from blank test solution (unit: g);

m -- Mass of the sample material (unit: g);

0.07408-- Coefficient for Quinoline phosphomolybdate to be converted into Na_3PO_4 ;

0.1718-- Coefficient for Quinoline phosphomolybdate to be converted into $\text{Na}_3\text{PO}_4\cdot 12\text{H}_2\text{O}$;

Take arithmetic mean of the parallel determination results as determination result, and the absolute difference between 2 parallel determination results shall not be more than 0.2%.

A.4.2 Volumetric method

A.4.2.1 Method summary

Add excess standard volumetric solution (VS) of HCl in the sample accurately, and use standard

VS of NaOH and titrate the excess standard VS of HCL; use a pH meter to indicate the jump point, and calculate the content of trisodium phosphate as per the consumption of VS of NaOH.

A.4.2.2 Reagent

A.4.2.2.1 Standard VS of HCL: $c(\text{HCL}) \approx 1\text{mol/l}$;

A.4.2.2.2 Standard VS of NaOH: $c(\text{NaOH}) \approx 1\text{mol/l}$.

A.4.2.3 Instrument and equipment

A.4.2.3.1 pH meter: is provided with glass electrode and saturated calomel electrode (or composite electrode), and the division value shall be 0.02pH unit;

A.4.2.3.2 Electromagnetic mixer

A.4.2.4 Analysis steps

Take and weigh about 2.7g~3.0g of sample of Na_3PO_4 or $\text{Na}_3\text{PO}_4 \cdot \text{H}_2\text{O}$ ignited as specified in A.11 ($\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ shall be 6~7g and be the original sample), and keep the accuracy to 0.0002g; put in a beaker of 400ml, and add 40ml of water without CO_2 to dissolve; use a pipette to add 50ml of standard VS of HCL accurately, and boil gradually and remove CO_2 and then cool it. Put the calibrate electrode of pH meter in the test solution, and use standard VS of NaOH for titrating the solution to a turning point occurring when pH value is about 4; record the consumed volume of standard VS of NaOH (V), and calculate the volume consumed standard VS of HCL (V1). Prevent that CO_2 is absorbed into the solution from the air, and continuously use standard VS of NaOH for titrating the solution to a turning point occurring when pH value is about 8.8, and record the volume of the consumed standard VS of NaOH (V2, i.e., the volume of titrated and consumed standard VS of NaOH from $\text{pH} \approx 4$ to $\text{pH} \approx 8.8$).

A.4.2.5 Calculation

The volume of the consumed standard VS of HCL when the test solution is titrated by standard VS of HCL and $\text{pH} \approx 4$ shall be calculated as follows (Formula A3):

$$V_1 = \frac{50c_1 - Vc_2}{c_1} \dots\dots\dots (\text{A.3})$$

In which:

V-- The volume of the consumed standard VS of NaOH when the test solution is titrated by standard VS of NaOH such that $\text{pH} \approx 4$ after 50ml standard VS of HCL is added and reacts in the test solution (unit: ml);

c1-- Accurate concentration of standard VS of HCL (unit: mol/l);

c2-- Accurate concentration of standard VS of NaOH (unit: mol/l).

The content of Na_3PO_4 or $\text{Na}_3\text{PO}_4 \cdot \text{H}_2\text{O}$ shall be calculated as per the mass fraction of the ignited dry basis of Na_3PO_4 (w1) and be expressed by % as shown in Formula A4 or A5:

$$\text{When } V_1c_1 \geq 2V_2c_2, \quad w_1 = \frac{V_2c_2 \times M_1}{1000m} \times 100 \dots\dots\dots (\text{A.4})$$

$$\text{When } V_1c_1 < 2V_2c_2, \quad w_1 = \frac{(V_1c_1 - V_2c_2) \times M_1}{1000m} \times 100 \dots\dots\dots (\text{A.5})$$

The content of $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$ shall be calculated as per the mass fraction (w2) and be expressed by % as shown in Formula A6 or A7:

$$\text{When } V_1c_1 \geq 2V_2c_2, \quad w_2 = \frac{V_2c_2 \times M_2}{1000m} \times 100 \dots\dots\dots (\text{A.6})$$

$$\text{When } V_1c_1 < 2V_2c_2, \quad w_2 = \frac{(V_1c_1 - V_2c_2) \times M_2}{1000m} \times 100 \dots\dots\dots (\text{A.7})$$

In which:

c1-- Accurate concentration of standard VS of HCL (unit: mol/l);

c2-- Accurate concentration of standard VS of NaOH (unit: mol/l).

V1-- The volume of the consumed standard VS of NaOH when the test solution is titrated by standard VS of HCL such that $\text{pH} \approx 4$ after 50ml standard VS of HCL is added in the test solution

(unit: ml);

V2-- The volume of the consumed standard VS of NaOH when the test solution is titrated by standard VS of NaOH and from pH \approx 4 to pH \approx 8 (unit: ml);

m -- Mass of the sample material (unit: g);

M1-- Molar mass of Na₃PO₄ (unit: g/mol, and M1=163.94);

M2-- Molar mass of Na₃PO₄•12H₂O (unit: g/mol, and M2=380.18);

Take arithmetic mean of the parallel determination results as determination result, and the absolute difference between 2 parallel determination results shall not be more than 0.2%.

A.5 Determination of pH value

A.5.1 Reagent

Water without CO₂

A.5.2 Instrument and equipment

pH meter: the division value shall be 0.02 pH unit.

A.5.3 Analysis steps

Connect the reference electrode and measuring electrode with pH meter, warm up and adjust to zero, and position it well.

Take and weigh 1.00g \pm 0.01g of the sample and put in a beaker of 100ml, dissolve with water without CO₂, and transfer to a measuring flask of 100ml; dilute to the scale and shake up. Pour into a dry beaker of 100ml, and use a pH meter to determine the pH value of the test solution.

A.6 Determination of content of arsenic

A.6.1 Reagent

A.6.1.1 Standard solution of arsenic: in 1ml of the solution, arsenic content (As) shall be 0.001mg;

Take out 1.00ml of standard solution of arsenic in accordance with the requirements of HG/T 3696.2 and put it in a measuring flask of 1000ml, dilute with water to the scale and shake up. This solution shall be prepared just before use.

A.6.1.2 Other reagents shall be prepared in accordance with Chapter 9 of GB/T 5009.76-2003.

A.6.2 Instrument and equipment

They shall be the same as in Chapter 10 of GB/T 5009.76-2003

A.6.3 Analysis steps

Take and weigh 1.00g \pm 0.01g and put in the measuring bottle of arsenic, and add 6ml of HCl to dissolve; and then operate in accordance with Chapter 11 of GB/T 5009.76-2003, i.e. "Add water to 30ml....." for the determination. The color of arsenic station on the mercuric bromide test paper shall not be deeper than the specified value in standard.

The standard shall be: use a pipette to take out 3ml of standard solution of arsenic, and treat it in the same time and same mode with the sample.

A.7 Determination of content of fluorine

A.7.1 Reagent

A.7.1.1 HCl solution: 1+11;

A.7.1.2 Sodium acetate solution: 3mol/l;

Take and weigh 204g of sodium acetate (CH₃COONa•3H₂O) and dissolve in 300ml of water, add 1mol/l of acetic acid solution and adjust the pH value to 7.0, and add water to dilute to 500ml.

A.7.1.3 Sodium citrate solution: 0.75mol/l;

Take and weigh 110g of sodium citrate (Na₃C₆H₅O₇•2H₂O) and dissolve it in 300ml of water , add 14ml of perchloric acid, and add water to dilute to 500ml.

A.7.1.4 Total ionic strength buffering agent

Sodium acetate solution (3mol/l) and sodium citrate solution (0.75mol/l) are mixed in equal volume, and it shall be prepared just before use.

A.7.1.5 Standard solution of fluorine: in 1ml of solution, the fluorine content (F) shall be 0.010mg.

Take out 1.00ml of standard solution of fluorine prepared in accordance with the requirements of HG/T 3696.2, and put it in a measuring flask of 100ml; dilute with water to the scale and shake up.

A.7.2 Instrument and equipment

They shall be the same as in Chapter 12 of GB/T 5009.18-2003.

A.7.3 Analysis steps

A.7.3.1

Take and weigh about 1g of the sample, and keep the accuracy to 0.0002g; put it in a beaker of 50ml, and add a small amount of water and then 10ml of HCl solution; after dissolving, transfer to a measuring flask of 50ml, and add 25ml of total ionic strength buffering agent, and add water to the and shake up for use.

A.7.3.2

Use a pipette to take out 1.00ml, 2.00ml, 4.00ml, and 8.00ml of standard solution of fluorine and put in measuring flasks of 500ml respectively, add 10ml of HCl solution and 25ml of total ionic strength buffering agent; add water to the scale and shake up for use.

A.7.3.3 Connect the fluorine electrode and saturated calomel electrode with the negative terminal and positive terminal of the measuring instrument, and insert the electrode into a plastic beaker with water, where there is a iron mixing rod with polyethylene bushing; during electromagnetic mixing, read the equilibrium potential value. After replacing the water for 2~3 times and when electric potential value is balanced, electric potential of the sample solution and standard solution may be determined.

A.7.3.4 Take the electric potential of electrode as ordinate and mass of fluorine (mg) as abscissa, and draw working curve on the semilog coordinate; and then find out the relevant mass of fluorine from the working curve of electric potential value of the sample.

A.7.4 Calculation

The content of fluorine shall be calculated as per the mass fraction (w_3) and be expressed by % as shown in Formula A8:

$$w_3 = \frac{m_1}{1000m} \times 100 \dots\dots\dots (A.8)$$

In which:

m_1 -- Mass of fluorine in test solution found out from the working curve (unit: mg);

m -- Mass of the sample material (unit: g)

Take arithmetic mean of the parallel determination results as determination result, and the absolute difference between 2 parallel determination results shall not be more than 0.0005%.

A.8 Determination of content of heavy metal

A.8.1 Reagent

A.8.1.1 HCl solution: 1+1;

A.8.1.2 Acetate buffer solution: pH \approx 3.5;

Take and weigh 25.0g of ammonium acetate and add 25ml to dissolve, and add 45ml of HCl solution; use diluted HCl or diluted ammonia to adjust the pH value to 3.5, and dilute to 100ml with water.

A.8.1.3 Hydrogen sulphide saturated water (prepared just before use);

A.8.1.4 Standard solution of Pb: in 1ml of solution, the content of Pb shall be 0.010mg;

Take out 1.00ml of standard solution of Pb in accordance with HG/T 3696.2 and put in a measuring flask of 100ml; dilute with water to the scale and shake up. This solution shall be prepared just before use.

A.8.1.5 Phenolphthalein indicating liquid: 10g/l.

A.8.2 Instrument and equipment

Colorimetric tube: 50ml.

A.8.3 Analysis steps

A.8.3.1 Preparation of test solution

Take and weigh $1.00\text{g} \pm 0.01\text{g}$ of the sample and put in a colorimetric tube of 50ml, add 20ml of water to dissolve, and then add 1 drop of phenolphthalein indicating liquid; use HCl solution to neutralize the solution to colorless, and then add 5ml of acetate buffer solution and 10ml of saturated hydrogen sulphide water; dilute with water to the scale and shake up. Put in shadow for 10min, observe it such that the color shall not be deeper than the standard colorimetric solution.

A.8.3.2 Preparation of standard colorimetric solution

Take out 1ml of standard solution of Pb and put it in a colorimetric tube of 50ml, and add 20ml of water; and then treat it as the same as colorimetric solution of sample in accordance with A.8.3.1, from "add 5ml of acetate buffer solution.....".

A.9 Determination of content of Pb

A.9.1 Reagent

A.9.1.1 4-methylpentanone-2 (MIBK);

A.9.1.2 Ammonia solution: 1+1;

A.9.1.3 Ammonium sulfate solution: 300g/l;

Take and weigh 30g of ammonium sulfate $[(\text{NH}_4)_2\text{SO}_4]$ and dissolve with water, and add water to 100ml.

A.9.1.4 Ammonium citrate solution: 250g/l;

Take and weigh 25g ammonium citrate and dissolve with water, and add water to 100ml.

A.9.1.5 Sodium diethyldithiocarbamate (DDTC) solution: 50g/l;

Take and weigh 5g of DDTC and dissolve with water, and add water to 100ml.

A.9.1.6 Standard solution of Pb: in 1ml of solution, the Pb content shall be 0.010mg;

Take out 1.00ml of standard solution of Pb in accordance with HG/T 3696.2 and put in a measuring flask of 100ml, and dilute with water to the scale and shake up.

A.9.1.7 Bromothymol blue indicating liquid: 1g/l;

A.9.1.8 Water in Class II: meet the requirements of GB/T 6682-2008.

A.9.2 Instrument and equipment

Atomic absorption spectrophotometer: with hollow cathode lamp

A.9.3 Analysis steps

A.9.3.1 Preparation of test solution

Take and weigh about 5g of the sample, and keep the accuracy to 0.01g; put it in a beaker of 150ml, and add 20ml of water; after dissolving, transfer to a extraction funnel of 125ml, and add water to 60ml. Add 2ml of ammonium citrate solution and 3~5 drops of bromothymol blue indicating liquid, and use ammonia solution to adjust pH value such that the solution becomes from yellow to blue; add 10ml of ammonium sulfate solution and 10ml of DDTC solution, and shake up. Keep for about 5min, and then add 10.0ml of MIBK and shake up intensely for extraction for 1min; keep in static state and form into layers, abandon the water layer, and put the MIBK layer into a graded tube 10ml with plug for use.

Simultaneously make blank test, during which no sample shall be added in the test solution, but others shall be the same, such as type and amount of reagent.

A.9.3.2 Preparation of standard reference solution

Use a pipette to take out 0.00ml, 2.00ml, 4.00ml, and 6.00ml of standard solution of Pb and put into extraction funnels of 125ml respectively, and treat it in the same time and same mode with the sample in accordance with the steps specified in A.9.3.1 from "add water to 60ml.....".

A.9.3.3 Determination

Adjust the instrument to the best operating state, and adjust to zero with water; inject the liquid extract as sample, and reduce the flow of acetylene gas appropriately. Determine the absorbance of standard solution, test solution, and blank test solution at the point of 283.3nm. Deduct the absorbance of zero standard solution from that of the standard solution, and take the mass of Pb (mg) as abscissa and the relevant absorbance as ordinate, to draw a working curve.

From the determined absorbance of test solution and blank test solution, find the mass of Pb

from the working curve.

A.9.4 Calculation

The content of Pb shall be calculated as per the mass fraction (w_4) and be expressed by % as shown in Formula A9:

$$w_4 = \frac{m_1 - m_0}{1000m} \times 100 \dots\dots\dots (A.9)$$

In which:

m_1 -- Mass of Pb in test solution found from the working curve (unit: mg);

m_0 -- Mass of Pb in blank test solution found from the working curve (unit: mg);

m -- Mass of the sample material (unit: g)

Take arithmetic mean of the parallel determination results as determination result, and the absolute difference between 2 parallel determination results shall not be more than 0.0001%.

A.10 Determination of content of water insoluble substance

A.10.1 Instrument and equipment

A.10.1.1 Sintered glass crucible: filter board aperture shall be $5\mu\text{m}\sim 15\mu\text{m}$;

A.10.1.2 Electrothermal constant temperature dry box: the controlled temperature shall be at $105^\circ\text{C}\pm 2^\circ\text{C}$.

A.10.2 Analysis steps

Take and weigh about 20g of the sample, and keep the accuracy to 0.01g; put it in a beaker of 400ml, and add 200ml of water and heat up to dissolve; use a sintered glass crucible keeping constant mass at $105^\circ\text{C}\pm 2^\circ\text{C}$ in advance for filtering, and wash with hot water till the filtrate is without alkalinity. Put the sintered glass crucible in an electrothermal constant temperature dry box of $105^\circ\text{C}\pm 2^\circ\text{C}$ to dry to constant mass.

A.10.3 Calculation

The content of water insoluble substance shall be calculated as per the mass fraction (w_5) and be expressed by % as shown in Formula A10:

$$w_5 = \frac{m_1 - m_0}{m} \times 100 \dots\dots\dots (A.10)$$

In which:

m_1 -- Mass of water insoluble substance and sintered glass crucible (unit: g);

m_0 -- Mass of sintered glass crucible (unit: g);

m -- Mass of the sample material (unit: g)

Take arithmetic mean of the parallel determination results as determination result, and the absolute difference between 2 parallel determination results shall not be more than 0.02%.

A.11 Determination of ignition loss

A.11.1 Instrument and equipment

A.11.1.1 Electrothermal constant temperature dry box: the controlled temperature shall be at $110^\circ\text{C}\pm 5^\circ\text{C}$;

A.11.1.2 High-temperature furnace: the controlled temperature shall be at $800^\circ\text{C}\pm 25^\circ\text{C}$;

A.11.1.3 Porcelain crucible

A.11.2 Analysis steps

Take and weigh about 5g of the sample, and keep the accuracy to 0.01g; put it in a porcelain crucible with constant mass at $800^\circ\text{C}\pm 25^\circ\text{C}$, and bake for 5h at $110^\circ\text{C}\pm 5^\circ\text{C}$; and then, transfer it to a high-temperature furnace of $800^\circ\text{C}\pm 25^\circ\text{C}$ and ignite for 30min, and cool in the drier to room temperature and weigh.

A.11.3 Calculation

The ignition loss shall be calculated as per the mass fraction (w_6) and be expressed by % as shown in Formula A11:

$$w_6 = \frac{m_1 - m_0}{m} \times 100 \dots\dots\dots (A.11)$$

In which:

m1-- Mass of the sample and porcelain crucible after ignition (unit: g)

m0-- Mass of porcelain crucible (unit: g)

m-- Mass of the sample material (unit: g)

Take arithmetic mean of the parallel determination results as determination result, and the absolute difference between 2 parallel determination results shall be: for Na₃PO₄, not more than 0.01%; for Na₃PO₄•H₂O, not more than 0.05%; for Na₃PO₄•12H₂O, not more than 0.1%.

END TRANSLATION